Notebook, N-03-10-05.2

F. L. Dyer

FRANK L. DYER
5 1903
ORANGE N.J.

Last week in September, 1903
Work on [illegible] patterns.

Clots [illegible]:

No. 1. $\bar{S}$ + Cer (80 + 20); when poured, wax 60°C, mould and core cold, chilling by wet rag with tap water (18°–20°C.) Smooth core taken out before mould taken off.

No. 2. $\bar{S}$ + Cer (80 + 20); when poured, wax 177°C. mould & core cold. Smooth core taken out as soon as possible.

No. 3. $\bar{S}$ + Cer (80 + 20), wax 177°C, mould & core hot. Smooth core taken out as soon as possible long before mould.

No. 4. $\bar{S}$ + Cer (60 + 40), wax 177°C, mould hot; smooth core taken out as soon as possible.

No 5. $\bar{S}$ + Cer (70 + 30); wax 177°C, mould & core hot; smooth core taken out

No. 6 $\bar{S}$ + Cer. (65 + 35); wax 177°C, mould & core hot, smooth core easily taken out.

No. 7 $\bar{S}$ + Cer (50 + 50), wax 177°C. mould & core hot; after [illegible] chilling, mould taken off, to take cylinder off.

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The threaded core, the cathode to be heated to about the temp. of the wax-

Results:

A. Cylinder Nos. 1, 2, 3, + 5 had a mottled appearance & showed cracking near under surface - ~~Hence~~ Nos. 4 + 6 had a uniform structure. Hence 65% S is the maximum

B. The ~~gold~~ mould used had a layer of gold, nevertheless, it was distinctly attacked at the high temperature of the casting, as shown by all the cylinders having a green layer ~~[illegible]~~ on the surface, due to copper attacked by the stearic acid-

C. Tap water (18°–20°C) was not cold enough to effect the shrinking out of the wax. In all cases it was necessary to ~~the~~ plunge ~~was~~ heated with wax into ice water (ca. 4°C) to take out cylinders. Even then the shrinkage was not great enough to allow of taking out the cylinders freely, a scratching against the mould being distinctly felt-

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D. Cylinders then tested on phonograph:

No 1. was found to have [illegible] in the [illegible] way, by mistake.

No. 2. Gave very scratchy sound and was very rapidly (by 3 or 4 reproductions) worn by the stylus — as distinctly seen under the microscope —

No. 3. Gave very scratchy sound and under the microscope showed rough record and wearing by stylus after 3 or 4 reproductions.

No. 4. The mould was found to have been filled in its upper part (nearer the end of core) and not filled in the lower half; the record was smooth where filled and rough where not filled. The reproducer left distinct marks of wearing after 4 reproductions.

No 5. The cylinder did not fill the mould, was irregular, and was rapidly worn by reproducing.

No 6. This was smooth, the mould was not filled, the record was partly destroyed by the heating necessary to get the core out. It was considerably worn

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by 4 reproductions.

Hence, Nos. 2–6 (i.e. all cylinders examined) were found to be extremely soft and to be worn by reproduction in 3–4 reproductions.

I. The melting-point of the commercial S used (Mitchell's) was found to be 56°C. Its solidifying-point was found to be 55.5

The melting-point of the ceresin used was found to be 52°.5–53°.5 C. Its solidifying-point was found to be 50°–52°.5 C.

Cylinder No. 6 was then examined as for alleged separation of its ingredients ≡ ~~([illegible])~~): a few milligrams were gently cut off the outside layer and the melting-point determined, it was found to melt gradually between 46° + 52°C. It was found to solidify very gradually on cooling between 47° and 43°C. A few milligrams were then scraped away from the inside of the cylinder mass (about half way in), the solid-

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fication-point was found to be 45°–43°C, no difference whatever being observed in the mode of solidification of this and of the outside layer.

It was therefore concluded, 1. that the outside layer and the mass of the cylinder were identical in composition, 2. that no separation and redistribution of the ingredients takes place, 3. that no such separation and re-distribution takes place at a temperature, as alleged, intermediate between the melting-point ~~and~~ or the solidification-point of the ingredients, both the melting-point and the solidifying-point of the mixture being, respectively, lower than those of either of the ingredients.

First Week in Oct, 1903 –

Further determinations of melting-points & sol.-points

No 4. Outside layer, Mp. 50°C. sharp
Solid.-p. 47°.5 – 44°C. (Solid looked non-homogeneous, because of slow cooling).

No 4. Inside layer. Mp. 50°C.
Solid.-p. 48°.0 – 44°.5 C.

No. 3. Outside layer. Mp: 49°–49°.5C. Solidif.-point. 46°–43°C. (perfect opacity ~~seen [illegible] at~~ only at a ~~too~~ little lower temp.).

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No. 3. Inside Layer. Mp. 51.5–53.5 C.
Solid-point: 51°.5–47° (~~White at~~ (turning white very slowly after that, white along bulb of therm. at about 40°C. ~~A~~ similar phenomenon, i.e. a second crystallization, must have taken place also in other cylinders examined, but was missed)—

No 5. Outside layer, mp. 51°–53°, Solid point: 49°–47.5 (Whiteness much lower)
No. 5. Inside layer

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M.P. determination [illegible] method:

30 g. S̄ + 20 g. Ceresin were melted in a glass beaker and the solidifying-point re-determined: the substance began to deposit crystals at 52.5 C., the temperature gradually fell to 45° C., remained stationary at this point, then rose pretty rapidly to 48.5 again remained stationary, then slowly fell to 47°, remained stationary (?), the mass being now in a pasty condition in the centre of the beaker and ~~pretty well~~ quite solid all round the sides. At 46.5 the temperature was stationary for a long time. When it was scarcely any longer possible to move the thermometer, the temperature was about 46.~~5~~C. - 45°C.

Further experiments, with superheating done in apparatus like the one described ~~b~~ in the patent; mixture used 65% Stearic acid and 35% ceresin.

Exp. 1. Apparatus superheated, then melted mixture poured in, heat maintained for ca. 15 minutes, then ~~a [illegible] of ordinary tapwater [illegible] maintained for ca. 30 minutes~~ rapid

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current of cold tap water turned on and maintained until the wax in the mould was solidified inwardly to within a very small distance from the core; mould, core, & wax then taken out, core removed and mould with wax replaced in the cooling jacket, after this the current of cold tap water was maintained for ca. 30 minutes; on careful examination, the wax showed no sign whatever of shrinking away from the mould. It was then kept in melting ice for about 15 minutes and then it was possible to remove the cylinder. The cylinder showed signs of powerful attack of the copper, being of a deep green hue. The cylinder is marked No. 8

Exp. 2. Now the mould & core were gently warmed; the wax was melted on the water bath and introduced into the mould. The mould, core, & wax were placed in the jacket, & the superheating & chilling were carried out as in the preceding experiment. The results were the same as before, except: a. the green

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coloration of the cylinder was not deep; b. the cylinder cracked longitudinally while the mould was in the melting ice. The cylinder is marked No. 9.

Exp. 3. The experiment was repeated for the purpose of obtaining a cylinder that was not cracked. This time there was irregular ~~of~~ cracking in several points, but the cylinder did not break open; it was kept for trial on the phonograph & microscopic inspection. It is marked No. 10. Cylinder No 10 was successfully reproduced.

Exp. 4. The ~~two~~ mould & core were then gently heated (to about the temp. of the mixture), the wax melted on the water bath & introduced into the mould. The latter was then chilled on the outside with ice water, the core taken out as early as possible, & the chilling continued until the wax shrunk away from the mould sufficiently for removing. The cylinder is marked No 11. (No jacket used)

Exp. 5. A smooth core was now employed

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and an ordinary mould (with thicker shell) was used. The preceding experiment was repeated, with the same result, except that it was a little more difficult to remove the cylinder, the top half of which cracked off irregularly. The cylinder is marked No. 12.

Exp. 6. The same experiment was repeated, with same result. The cylinder is marked No. 13.

Exp. 7. A cast was now made with the regular white-blank composition. The mould and core were heated by a flame, the wax was heated until limpid, introduced into the mould, & ~~Ea~~ as soon as possible the core was removed; on the application of ordinary tap water to the mould, the wax soon shrank away & was removed. The cylinder is marked No. 14

Exp. 8. Exp (5) was repeated, again with threaded core and thin mould. Cylinder shrunk away from the mould pretty easily. The cylinder marked No 15.

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Exp. 9. Experiment (5) was again repeated with threaded core. The cylinder came out entire, without any cracking. It is marked No. 16

Exp. 10. Experiment (5) was repeated with a mixture of 75 parts of ceresin and 25 parts of stearic acid. The cylinder came out entire. It is marked No. 17

Exp. 11. A mixture of 65 parts of stearic acid and 35 parts of ceresin were heated at 177° C. (350° F.): no bubbles were given off.

Microscopic Examination of Cylinders 7 — 17 by Mr. Edison Oct. 12, 1903, who said:

No. 7. One part of record fair, other part dull, torn, rough, apparently not filled seems as if gas formed on surface in places. The record has a blue tinge also is cracked.

No. 8. Record very green; lots of gold torn off the mould; surface dull, don't seem

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to be well filled; part of a cavity shiny part dull; looks as if surface was torn.

No. 9. Part of record very blue; cracked whole length; very dull surface; don't look as if it filled anywhere; very poor.

No. 10. Cracked; very bad; dull surface generally; very blue; gold torn off; large patches of record torn and rough; very poor.

No. 11. Some of gold torn off; surface rough; not shiny; has torn appearance over large area; cracked; very bad record; blue tinge.

No. 12. Shows gold torn from mould on bottom end; longitudinal scratches from removing and from mould; surface generally dull; pretty well filled; crack in record.

No. 13. Record with cracks; some torn parts in it; not very shiny; seems pretty well filled; some places show air or gas

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caught

No. 14. Gold torn off mould slightly; a little dull at top end; fills pretty well; looks fair under microscope.

No 15. Cracks in spots; bad surface; torn; not filled in places.

No. 16. Dull surface; green or blue tinge; some parts of area shiny, others dull, with torn appearance; a few specks of gold torn off; cylinder intact, but cracked.

No. 17. Crystallized or torn and rough surface, does not seem to be filled; dull on one side, brighter on the other side in places; chipped, probably stuck to mould in places. Top pouring end very bad surface, bottom better; fairly shiny, but surface torn. One inch from bottom the surface is not shiny and is torn.

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60 g. $\bar{S}$ + 40 g. Ceresin melted, thoroughly mixed, a sample sucked up into a capillary tube, + mp determined; result: 49°-50°C. There may have been incipient melting somewhat

Observation: The mixture was introduced (melted) into ~~alum~~ gently warmed mould allowed to solidify & then subjected to superheating (i.e. No 18). Same of No. 19.

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below 49°C., but it only became distinct at 49°C – Solid. p. 45°.5–44°C (in capillary tube) The solid-point of the same mass in a beaker was as follows: first depositing of crystals at 51.5 or 52°, then gradual fall of temperature to 45°, then gradual rise to almost 49°, remained long stationary at 48.5°, then temperature of paste-like mass gradually fell to about 45°C. The result was thus identical with the one described on p. 7.

+ No 18: DA mixture ~~[illegible] in Exp 11.~~ ~~(p. 11)~~ was cast in ~~[illegible]~~ mould (4917) heated in glycerin bath to 177°C for some time & then plunged into cold water. Smooth core was closed at the bottom with a cork stopper. The composition of the mixture was ~~[illegible]~~ that (65:35) that had been used in Exp 11 (p. 11). The heating was stopped before all bubbling ceased.

No 19. 91 g S + 49 Ger. (= 65:35) were heated as in preceding Experiment, but until all bubbling had ceased. The

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core was left open, so that both superheating & chilling were done both from the inside and from the outside. After this the mould was carefully examined under the microscope: it was found to have been badly attacked by the wax.

Analysis:

Residue of wax ~~I~~ from casting No. 19 was melted up and two samples taken in the liquid state, from well mixed mass.

1. Weight of sample A .. 1.0370 g.
2. " " " B .. 1.0095 g.

A thin outermost layer was then scraped away from cylinder No. 19, and weighed as 'sample C'.

3. Weight of sample C .. 0.7880 g.

Finally, as a check, a sample of stearic acid was taken (Sample D).

4. Weight of sample D .. 0.9935

~~To ea~~ The four samples were then

titrated in alcoholic solution with a ca. 1/3 N. alcoholic solution of KOH and phenol phthalein as an indicator. The numbers of cubic centimeters of alkali required to neutralize the four samples were as follows:

5. Sample A . . . . 7.85 cc.
∴ 1g. A = 7.59 cc. alkali
6. Sample B . . . . 7.72 cc.
∴ 1g. B = 7.65 cc. alkali
Average:
1 g. wax = 7.62 c.c. alkali

7. Sample C . . . . 6.00 cc.
∴ 1 g. C = 7.61 cc. alkali

8. Sample D . . . . 11.64 cc.
∴ 1g. D = 11.72 cc. alkali

7.62 is 65 per cent. of 11.72.

Conclusions: 1. The analytical method used is very good and would easily show a difference of 1%; 2. The outside layer of the cylinder is identical in composition.

with the mixture employed in casting the cylinder, and hence a separation of the ingredients is seen not to take place.

Determination of ~~Con~~ Point at which a film can form on the mould.

1, Glass apparatus used (thin walls) with cold water circulating through it and a thermometer attached at ca. 1/16 of an inch. A number of determinations (about 12) gave on an average:
a. Temperature at which film ~~is~~ covers about ½ in. of the glass tube from the bottom : ca. 93°C. (200°F.)
b. Temp. at which film covers entire length immersed in molten wax (viz. 1½ in.) ... ca 65°C. (150°F.)
c. When stirring was employed, the temp. at which ½ in. from bottom was covered with a film was ca. 75°C. (167°F.)

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2. Metal apparatus employed, consisting of a ~~tub~~ brass tube (1/16 in. thick) with water circulating through it and a copper ring about an inch tall and made of 1/16 in. of copper, fitting snugly around the brass tube. In this the conditions are very similar to those of the apparatus described in the patent; the cooling is, on account of smaller dimensions, probably more efficient.

With this apparatus, the ~~maximum~~ temperature at a distance of ca. 1/16 in. from the copper ring was found to be ca. 120°C. (248°F.) when the first deposit was formed on copper ring. Above this temp. there was not a trace of deposit formed.

Cylinders for ~~trying~~ testing hardness and resistance to wear by stylus:

No. 20. Commercial brown wax, ~~with~~ mechanically reproduced record.

Wax of Nos. 26—35 has been heated to 200°C. before casting, for the purpose of driving off any impurities that might be present.
Comp.: 65:35 ($\bar{S}$ + Cer.)

No. 21. Same as No. 20.

No. 22. Brown wax cast, when liquid, in gently warmed mould and subjected to very slow cooling.

No. 23. Brown wax maintained in glycerin bath at 177°C. for ca. 10 minutes then suddenly chilled from outside by plunging into icewater.

No. 24 Blank of brown wax made by Mr. Dodd in the ordinary way.

No. 25. Same as No. 24.

Experiment. Mixture 65 $\bar{S}$ + 35 Cer. were filtered at 100°C. through Swedish filter-paper.

No. 26. $\bar{S}$ + Cer. (65:35) ~~at~~ cast warm, allowed to cool slowly; when perfectly solid, but somewhat warm, chilled out of mould with ice.

No. 27. $\bar{S}$ + Cer. (65:35), cast at gentle warmth, allowed to cool slowly; when solid & quite cold, chilled out of mould with ice

No. 28. Sudden chilling from about 60°C.; water not very cold; when wax [illegible] cold, chilled out with ice.

No. 29, Sudden chilling from about 60°C, Ice water.

No. 30 Sudden chilling from about 100°C. Ice water

No. 31. Sudden chilling from about 175°C. Ice water

No. 32. Sudden chilling from about 175°C. Ice water

No. 33. Slow chilling from about 100°C.

No. 34 Sudden chilling from melting point. Ice water.

No. 35. Sudden chilling with ice water from melting point.

No. 36. Regular white wax,
suddenly chilled from about 175°C.

Hardness of Mixture of 65 S + 35 Cer. cast into blanks in different ways: & tested with constant weight re-corder. The width of the groove, approximately ~~was~~ estimated by inspection under the microscope, was as follows:

1. Cyl. made by slow chilling,
average of 3 cylind. . . . .0080 in.

2. " " sudden chilling from
60°C., average of 4 cyl. . . .0078 in

3. " " by sudden chilling from 100°C. .0075 in

4 " " sudden " from 175°C
(average of 2 cyl.) . . .0078

Hardness of ordinary compositions:

1. Reg. white made by slow chilling . . .0060

2. " " " " sudden chilling
from 175°C. . — — . . .0060

3. Reg. brown made commercially 0060
4 " " " by slow chilling .0060
5. " " " by chilling from 175°C .0080(?)

Conclusion: Mixtures of stearic acid and ceresin are softer than ordinary waxes, and their hardness is not influenced by sudden chilling.

Brown commercial cylinder cast by process described in Macdonald patent ~~was~~ examined as to separation of ingredients (Cylinder No. 2-3):

Weight of Outside layer . . . 1.0720g.
" " inside " 1.1805

No. of cc. of alcoholic KOH required to neutralize outside . . . 5.15 cc.
Same — inside . . . . 5.50 cc.
∴ 1 g. outside requires 4.80 cc.
∴ 1 g. inside " 4.65

1 g. commercial stearic acid requires 11.72 cc.
∴ Outside contains 40.9% } Practically
Inside " 39.6% } the same.

Attempt to analyze the commercial cylinders made by the Columbia Graphophone Company. – Nov. 1, 1903 –

Sample No. 1, weight . . . 10.0050
(very slight loss occurred at an early stage of the ~~p~~ analysis).

Sample No. 2, weight . . . 10.0045+
[illegible]
[illegible]

(1) Wt. of Pt. cruc. + lid 50.4780
(2) Wt. of (1) + Al_2O_3 . . . . . 50.5205

Al_2O_3 .0425 g.
~~Al~~ or 0.425%

(1) Wt. of Pt Cruc. + lid 50.4775

Coal

Pt cruc.	50.4775
+ sample (4935)	51.9300
sample	1.4525
Cruc. + res. (1st weighing)	50.7515
(2d ")	50.7515
Res.	.2740 (18.9%)
~~Porc. cruc. + lid~~	~~23.7680~~
~~+ sample (combined)~~	~~25.5710~~
~~sample~~	~~1.8030~~
Porc. cruc.	13.8110 − − 200
+ sample	15.3010 − − 200
Sample	1.4900
+ res. (1st weighing)	14.1025 − − 200
Res.	.2915 (19.6%)
+ res. (2nd weighing + HNO₃)	14.1000 (19.4%)

Mean: 19.5%

[ITEM FOUND IN BOOK]

One part fair other
part dull torn, rough
apparently not filled
Seems as if gas formed a
[illegible]
Record blue tinge,
Cracked –

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7 = Record very green
Lots of gold torn off mould
surface dull – dont seem
to be well filled – part of
a cavity shiny part dull
looks as if surface Torn

[ITEM FOUND IN BOOK]

9 = part of Record —
Very blue — Cracked color
length — very dull
surface dark [illegible]
if it filled Everywhere
very poor — [illegible]

10 — Cracked — very bad
dull surface generally
Very blue — gold torn off
large patches [illegible] record
torn rough — Very poor

[ITEM FOUND IN BOOK]

11 — Some of gold torn off
Surface rough not shiny
has torn appearance over
large area — Cracked
Very bad record —
On a part of record grooves
in blue tinge —

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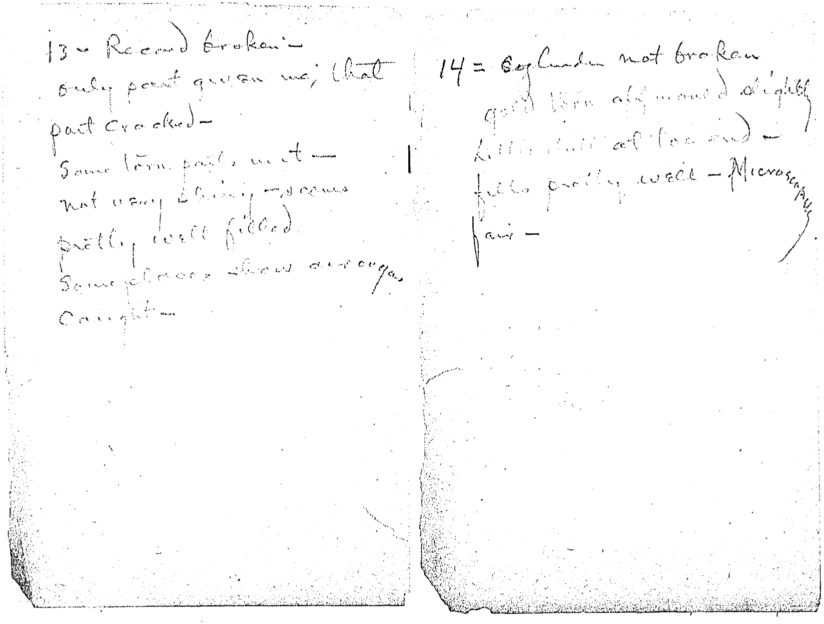
12 = Shows gold torn from
mould on bottom and
Longitudinal scratches from
removing of from mould
Surface generally dull
pretty well filled —
Record broken only part
given me — also Crack
in part given

[ITEM FOUND IN BOOK]



13 – Record broken –
only point given me; that
part cracked –
Some form parts wet –
not very shiny – seems
pretty well filled.
Some places show air [illegible]
caught –

14 = Cylinder not broken
good form of mould slightly
little dull at the end –
fills pretty well – Microscopy
fair –

[ITEM FOUND IN BOOK]

15 – Only part of record
remaining
given me – Cracked in
spots – bad surface –
torn also, not filled in
places

16 = Crystalized or dull surface
green or bluish tinge –
Some areas shiny others
dull with torn appearance
few spots gold torn off –
Cylinder intact but
cracked –

[ITEM FOUND IN BOOK]

17 = Crystalized surface generally
or may be due to torn surface
Rough surface
Dont seem to be filled
Dull on one side brighter on
other side in places
Chipped - probably stuck
to mould in places
Top pouring end very bad
surface - bottom better
fairly shining but surface
torn one inch from bottom
matching surface torn